Comparative Analysis of Methodology of Particle Size Analysis

An Honors Thesis (HONR 499)

By

Kelsey West

Thesis Advisor

Dr. Jessique Haeft

Ball State University
Muncie, Indiana

December 2018

Expected Date of Graduation

December 2018

Abstract

The analysis of the composition of soils proves useful for a variety of individuals, even outside of the academic community. Architects, farmers, and others may use the determined soil type to determine materials, layouts, and crops that will work best in certain areas based upon the manner in which the soil interacts with water and other nutrients. The way in which scientists determine the composition of soils is known as Particle Size Analysis (PSA). I comparatively analyze two different methodologies known as the Bouyoucos (Hydrometer) method and the Pipette method. Through this analysis I explore the laws which these methods are based upon and how the differences in methodology may impact which is chosen when analyzing soil samples.

Acknowledgments

I would like to thank Dr. Jessique Haeft for advising me through this project. I have greatly appreciated her help with the research and writing of this paper.

I would also like to thank my family and friends who continuously encouraged me throughout this entire process.

Process Analysis Statement

The aim of this project was to analyze the information that I have learned regarding particle size analysis over the past year and better understand how the various methodologies differ from one another. I am a science major and as such complete experiments and am familiar with basic laboratory techniques. Therefore, it makes sense that my thesis revolves around such techniques in a practical setting. I was introduced to particle size analysis and Dr. Haeft during my undergraduate honors fellowship. The research completed during this time helped to introduce me to the pipette method, and I was curious as to how it differed from the hydrometer method.

For my project, I was mostly interested in the differences between the two methodologies and how such differences could impact which method researchers chose to utilize in their experiments. The majority of my project was based heavily upon research. I had some experience with the pipette method and therefore was able to use my experience to drive my writing; however, the hydrometer method was something I had never before encountered. From the beginning the research seemed to have a natural flow. I knew that I wanted my paper to be organized in a manner that introduced particle size analysis as a whole, talked about the laws governing such a process, and then get into discussions of the two different methods. Dr. Haeft was extremely helpful in pointing me in the direction of resources that contained much of the information that I needed, more specifically the books that contain the basics of the methods. It was a bit more work to find the papers that discussed the first experiments done in the development of the methodologies. Some of the papers were published in the 1920's and had to be requested through Interlibrary Loan in order to have the information. Once I was able to access the beginning research, the changes made to the methods was easier to find through Ball

State's OneSearch/Cardcat system. I also was able to utilize Ball State's libraries in another manner as I found books that in depth discussed soil composition and the laws governing analysis of such soils. In order to find some of the information regarding price points of the necessary equipment, I had to ask the Lab/Field Instrumentation Technician of the Department of Natural Resources to access the websites of the companies. Many of them require a password in order to access such information and I was not able to obtain one on my own.

Through this process I found that I have learned a great deal while at Ball State that has changed how I approach research. I was able to find the resources I needed in an efficient manner even if I had to wait a few weeks before I was able to access those resources. I also learned a great deal about soil compositions. While I had learned quite a bit through my fellowship, this project greatly expanded upon my knowledge and helped me to better analyze how I could have completed my experiments differently.

Particle Size Analysis (PSA)

Particle size analysis (PSA) encompasses the laboratory techniques that are used to determine the size distribution of particles that are found within a soil sample. Soil aggregates are broken down and particle distribution determination is done using the known size limits of texture classes. Texture classes, and the distribution amongst these classes, are used to determine the soil type of that which was analyzed. Sand, silt, and clay are the texture classes of known size limits that are used for such determinations.

Soil aggregates are separated into individual soil particles with ultrasonic, mechanical or chemical methods (Dane et al., 2002) using techniques such as sieving, sedimentation, and others. During the sieving process, soil samples are passed through a series of coarse screens with specific openings (Jury & Horton, 2004). The sizes of the remaining dispersed particles are then characterized through sedimentation processes, typically either the hydrometer method or pipette method. The discrete units that are collected are the individual texture classes and are analyzed to determine the percentages of each that are present. The size determinations used differ based on the specific classifications that are used. Those used by the U.S. Department of Agriculture (USDA) are as follows (Garcia-Gaines & Frankenstein, 2015):

Clay: less than 0.002 mm

Silt: 0.002-0.05 mm

Sand: 0.05-2 mm

The known size differences are used to determine the best method for separation of the particles based upon available equipment and known methodologies. The determination of soil type that is decided upon based upon these size distributions can be further used by other individuals.

Architects, farmers, and others may use the determined soil type to determine materials, layouts, and crops that will work best in certain areas. Each soil type interacts with water and other materials in a unique manner that must be understood for the areas to be useful to those utilizing them. Clay is composed of the smallest sized particles and as such, the particles cling together. This clinging together of particles prohibits water and nutrients to readily move through soils that are composed of high percentages of clay (Väderstad Group). Clay fractions of soil contain Si, Al, Fe, H, and O primarily with smaller amounts of Ti, Ca, Mg, Mn, K, Na, and P (Jury & Horton, 2004). The presence of these elements, as well as the quantities present, also impact the ideal way to interact with the soil, especially for those in an agricultural industry. Sand particles are quite coarse and this allows water and nutrients to leach rapidly to particles. Depending on how the areas high in sand content are proposed to be used, this leaching could occur too rapidly and not allow water and nutrients to be moved in an appropriate manner (Väderstad Group). Silt contains particles that are at size limits between those of clay and sand. Thus, it is easily compacted and often makes up the fertile aspect of soils (Väderstad Group). Sand and silt contain many primary minerals that have impacts upon soil weathering and development (Jury & Horton, 2004). Overall, sand and silt have small specific surface areas and therefore have a minor influence upon chemical and physical properties of soils (Jury & Horton, 2004). The two most common methods for particle size analysis are the Bouyoucos, or hydrometer, and Pipette methods. Both methods depend fundamentally upon Stoke's Law.

Stokes' Law

Stokes' law establishes a direct relationship between the rate of sedimentation and particle size; thus, allowing the settling velocities of particles to be used to quantify particle size. Gravitation, buoyancy, and viscous drag are the three forces that work upon spherical particles as the fall through a liquid (Jury & Horton, 2004). While soil particles are not exact spheres, the assumption is made that they act in the same manner in order to quantify the impacting forces. The forces will depend upon the particle's density and radius, as well as the density and viscosity of the liquid into which it is placed (Jury & Horton, 2004). Newton's Law allows for the calculation of the gravitational force through the following equation:

$$F_g = m_s g = \rho_s V_s g = \rho_s \frac{4\pi R^3}{3} g \qquad (1)$$

Where m_s is the mass of the particle and V_s is the corresponding volume of the particle (Jury & Horton, 2004). The gravitational force works in a downward manner, therefore, it pulls the soil particles toward the bottom of the container. Archimedes' principle allows for the calculation of the buoyancy force through the following equation:

$$F_b = m_l g = \rho_l \frac{4\pi R^3}{3} g \qquad (2)$$

Where m_l is the liquid mass that is displaced by the volume of the solid (Jury & Horton, 2004). This force acts in an upward manner upon the particles, pushing them in the opposite direction of the gravitational force. If the soil samples are evaluated through experimental procedures using the same liquid, then it is assumed that both the buoyancy and gravitational forces will remain constant across all samples. Therefore, the viscous drag force will be the force that has the greatest impact upon the results.

Stokes' Law allows for the calculation of viscous drag felt by the particle as it falls with a particular velocity through the following equation:

$$F_d = 6\pi R \eta V \qquad (3)$$

This force acts in a manner that is opposite of the velocity; thus, for a particle falling downward through a liquid, the force acts in an upward manner (Jury & Horton, 2004).

The three forces can be combined in order to determine the net force acting upon the particles in the suspension through the following equation (Jury & Horton, 2004):

$$\sum F_i = 0 = F_g - F_l - F_d \tag{4}$$

When Equations 1, 2, and 3 are then inserted into Equation 4, the velocity can be solved for in the following manner (Jury & Horton, 2004):

$$V = \frac{(\rho_s - \rho_l)\, D^2 g}{18\eta} \tag{5}$$

A solution containing mixed suspension of particles of various diameters will then have particles that settle to the bottom of the container at various rates. The hydrometer method then uses a pre-calibrated floating object which sinks lower in the solution based on the density of the displaced solution (Jury & Horton, 2004). The placement of this object at any given time can then be used to calculate the density of the solution and the mass of the particles within a given size range (Jury & Horton, 2004). The other approach, the pipette method, involves "direct sampling of the solution in suspension at various times (Jury & Horton, 2004)". Both methods make four basic assumptions when applying Stokes' Law which are the following:

"1. Terminal velocity is attained as soon as settling begins.

2. Resistance to settling is entirely due to the viscosity of the fluid.

3. Particles are smooth and spherical.

4. There is no interaction between individual particles in the solution. (Dane et al., 2002)"

These four assumptions allow for direct application of Stokes' Law without having to include other equations to account for particle interactions. These assumptions indicate that such interactions have a negligible effect upon the final outcome of analysis.

Bouyoucos (Hydrometer) Method

The hydrometer method was developed by Georgy Bouyoucos in 1927 and was then improved upon by Bouyoucos in 1962. The idea was to measure the rate of settling of soil particles and use the data collected to obtain a distribution curve (Bouyoucos, 1927). The formed distribution curve would then allow for the calculation of the size distribution of soil particles (Bouyoucos, 1927). The hydrometer that was used in 1927 was a Zuevenne's Lactodensimeter with a large surface volume and considerable weight, leading it be sensitive and accurate in the results obtained (Bouyoucos, 1927). A calibration of the hydrometer was done in order to allow for direct reading of densities (Bouyoucos, 1927). Varying concentrations of suspensions were done and drying of the suspensions allowed for the determination of the amount of dry material present within the samples prior to anlysis. The method was completed at room temperature and in the way described by Bouyoucos, which was done in the following fashion:

1. 75 grams of soil were placed into a mortar and soil was washed two or three times with distilled water.
2. Soils were rubbed with pestle to break up soil particles and disperse the particles.

3. Soil was mixed with water and allowed to stand for a few seconds before the supernatant liquid was poured into a large cylinder (approximately 1100 cc capacity).
4. Steps 1-3 were repeated until all the soil material was dispersed. The cylinder was then filled with distilled water and shaken for about five minutes.
5. The hydrometer was placed into the suspension and readings were completed every minute. The readings were continued in this manner for various amounts of time, with the rate of settling becoming quite slow after the first hour.

The hydrometer used for the experiment gave an average of the densities present in the column at all depths. The collected data can then be used to obtain an accumulation curve where diameter is based upon Stokes' equation (Equation (3)). When Bouyoucos made improvements upon his method in 1962 a small change was made to the procedure above. The original method indicated that soil should be soaked overnight and then stirred for 6 to 25 minutes (Bouyoucos, 1927). The improvement made upon this meant that the soil was soaked for 15 to 20 hours and then stirred for approximately 2 minutes (Bouyoucos, 1962). The soaking still occurred in the same solution of 5% Calgon in water (Bouyoucos, 1962).

Today, the method has somewhat been improved upon with Bouyoucos still holding claim to the ideas behind the largest aspects of the Hydrometer Method. Calibration of the hydrometer must be done prior to any other steps of the process. This is done by adding 100 mL of HMP solution with enough distilled water to reach a volume of 1 L in a cylinder, the solution is mixed, and a determination of the temperature is completed. The hydrometer is lowered in order to determine the hydrometer reading of the "blank" solution prior to any soil being introduced in order to determine the correction used during analysis of samples (Dane et al., 2002). Once the hydrometer has been calibrated, the soil must be dispersed. A sample of soil is weighed into a beaker with 250 mL of distilled water and 100 mL of HMP solution also added. This is soaked overnight while another sample is dried overnight at 105°C to yield the over-dry weight used during analysis. The treated sample is transferred and allowed to shake overnight in a horizontal shaker. From there the sample is added to a cylinder with enough distilled water to reach the 1 L volume marking (Dane et al., 2002). The solution in the cylinder is mixed and the hydrometer immediately lowered where readings are recorded at thirty seconds and one minute (Dane et al., 2002). The times of the readings following the initial ones are decided based on the individual, with the hydrometer being removed, rinsed, dried, and reinserted ten seconds prior to each reading. As the hydrometer does not give results regarding the sand fraction of the sample, a separation of these particles must be completed. This is completed through the transfer of the sediment in the cylinder through sieves corresponding to the known size of sand fractions. The sieve used may vary based on the brand of sieves used and the known values given by the USDA should be referenced in order to determine the appropriate sieve. The sediment is washed using a wash bottle filled with distilled water. The collected sand is moved to a tared beaker, dried overnight at 105°C, and weighed (Dane et al., 2002). The data collected is then analyzed to determine the percentages of clay, sand, and silt present within the tested samples.

The hydrometer method, as mentioned, relies upon Stokes' equation for the determination of particle size within samples. For the hydrometer method, the equation is used in the form:

$$\Theta = \left(\frac{18\eta h'}{[g(\rho_s - \rho_l)]}\right)^{1/2} \tag{6}$$

Where h' is the hydrometer settling depth given in centimeters (Klute, 1986). The settling depth of the particles can then be approximated using hydrometer dimensions using the following equation:

$$h' = L_1 + \frac{1}{2}\left(L_2 - \frac{V_n}{A}\right) \quad (7)$$

where L_1 is the distance along the hydrometer stem, L_2 is the overall hydrometer bulb length, V_n is the volume of the bulb, and A is the sedimentation cylinder's cross sectional area (Klute, 1986).

Pipette Method

The pipette method involves a more direct sampling than the hydrometer method. A small sample is taken from the larger sample by a pipette at a designated depth based upon the amount of time the solution has been allowed to settle. In direct contrast to the hydrometer method, the more coarse particles are removed prior to the sampling taking place. The pipette method of analysis has been believed to have been developed in three different countries simultaneously in 1922 (Black, 1965). The method described here is that used by Gee and Bauder in 1986 (Klute, 1986).

As mentioned, the more coarse particles are removed first; thus, the sand fraction is removed prior to the suspension being created in the 1 L cylinder. The night prior a sample of soil is added to a shaker bottle along with 400 mL of distilled water and 10 mL of HMP solution (Dane et al., 2002). This solution is shaken overnight in a horizontal shaker. The solution is then poured through a sieve into a 1L cylinder. The sieve is once again determined based on the brand of sieves used in the laboratory with reference to the known sizes of particles given by the USDA. The fraction collected on the sieve is then transferred to a tared beaker, dried overnight at 105°C, and weighed (Dane et al., 2002). Following drying of the sand, the fraction is sent through a series of sieves arranged in decreasing size, shaken on a sieve shaker for three minutes, and each sand fraction is weighed (Dane et al., 2002). The fraction collected within the sedimentation cylinder used to determine the percentage of clay and silt present in the sample. The cylinder must be filled to the 1 L mark with distilled water and allowed to stand for several hours untouched to allow for equilibration. Following the elapse of time, the pipette is lowered to the appropriate depth within the solution based upon the temperature of the solution and the time elapsed. The pipette withdraws a 25 mL sample and the sample is placed into a tared dish, dried overnight at 105°C, cooled in a desiccator, and weighed (Dane et al., 2002). The data collected is then analyzed to determine the percentages of clay, sand, and silt present within the tested samples.

The pipette method, as mentioned, relies upon Stokes' equation for the determination of particle size within samples. For the pipette method, the equation is used in the form:

$$t = (\frac{18\eta h}{[g(\rho_s - \rho_l)X^2]} \qquad (8)$$

Where h is the depth the pipette is lowered given in centimeters (Klute, 1986). Klute (1986) mentions that "settling times for the clay fraction can be calculated for sampling at a given depth for a given temperature". The use of HMP solutions also must be taken into account when determining the depths used a various temperatures due to changes caused by the solution viscosity and density. The following relationship has been found to exist:

$$\rho_l = \rho^{\circ}(1 + 0.630\ C_s) \qquad (9)$$

Where ρ_l is the solution density at a certain temperature, ρ° is the water density at the same temperature, and C_s is the concentration of HMP (Klute, 1986). Another important relationship that closely corresponds to Equation (9) has been found to be (Klute, 1986):

$$\eta = \eta^{\circ} (1 + 4.25\ C_s) \qquad (10)$$

Advantages/Disadvantages

Both of these methods can be used relatively easily within lab settings. Other than basic lab techniques that are required for both procedures and understanding of the equations/calculations that must be done, both are fairly straight-forward and many individuals could carry experiments to completion. The understanding of the results would bring about a need for a deeper understanding of the implications of soil textures and percentages of fractions of clay, silt, and sand found; however, this understanding would not be needed during the carrying out of the procedures. The results found by both the hydrometer and pipette method have been found to be significantly correlated; thus, either procedure should bring the individual to the relatively same conclusion regarding the soil that is being sampled (Miroslaw et al., 2014).

The use of the pipette method could be considered more simple based on the fact that it is a direct sampling. The individual operating the pipette would determine the accuracy of the results based on how precise they were in going to the proper depth. Also, the error incurred throughout the process would be human error without any correction factor or calibration being needed. This could also be looked at as a downside to the method as well. The hydrometer method does allow for the correction factor which could make small changes insignificant, while the results obtained through the pipette method are used as accurate without any leeway for error.

However, for the hydrometer method the human error that could occur through improper calibration of the hydrometer itself could be significant. While slight mis-calibration would be corrected for based upon the use of the blank as a baseline, trying to compare the results obtained from these samples to others could result in discrepancies and further problems.

The pipette method also requires only one sampling of the solution in the cylinder, rather than a series of samples taken over an extended period of time. In some aspects, this is an advantage for the method as it requires an individual to disturb the solution only once negating effects that could arise from faster sedimentation from the ripples created from the lowering of the hydrometer each time it enters the cylinder. This is also a disadvantage as it only gives one data point for the entire time that the solution is in the cylinder. The data that is obtained must be used for calculations even if unknown errors occurred and skew the results. The skewing of data could be negated to some degree through the use of multiple samples being taken of the same larger soil sample. The use of multiple data points to determine the percentage of clay and silt arises from the use of the hydrometer method with multiple data points being collected over an extended period of time. While the pipette method could display skewed data to a greater degree due to the collection of only one data point, the collection of multiple data points could help to negate any points that are outside of the "norm" for the sample.

Both methods require an extended amount of time to be put into them prior to calculations being done, although the time spent is not in the same manner. The pipette method requires the sample to shake overnight and for the suspension solution to sit for several hours prior to the sample being taken from the cylinder. The hours can then be spent by the researcher in a variety of ways. They could choose to stay in the lab, or they have the option to leave and come back to remove a small sample when the equilibration has occurred. The hydrometer method also requires the sample to shake overnight, but then the extended period of time spent comes from the collection of multiple samples. The samples are taken at 3, 10, 30, 60, 90, 120, and 1440 minutes (Klute, 1986). The samples taken between 3-120 minutes may require the researcher to stay in the lab during this time to ensure that the samples are collected at the appropriate times. The sample taken 24 hours later would also need to be done at the appropriate time, but would not require the individual to stay in the lab during that entire amount of time.

The hydrometer and pipette methods also require that the fractions be separated, although the timing of this separation is different between the two methods. The separation during the hydrometer method takes place following the determination of the clay fraction using the hydrometer. The removal of the sand requires the passing of the sample through only one sieve which is an advantage for this method. A downside to this is that it simply allows for the calculation of how much sand is within the sample without furthering that distribution into various sand coarseness. The pipette method requires the separation of the fractions to occur prior to the transfer of the clay portion to the cylinder. The sample is passed through a sieve and the sand is collected. However, following the drying of the sample, the sand portion is passed through a series of sieves that vary in size. While this makes the process more time consuming, it does allow for the individual to determine to a greater degree the exact distribution of sand present within each sample that is tested.

An advantage with the pipette method is the relatively small amount of sample needed. The method requires between 5-25 grams of sample to be used each time to run the analysis of the soil (Coates & Hulse, 1985). While the hydrometer method does not require significantly more than that, it does require 30-40 grams of sample each time (Coates & Hulse, 1985). The amount of sample is not larger in either case, but if only a small amount of sample is present, and samples must be run in duplicate, it may be advantageous to use the method that requires the least amount of sample to obtain accurate results.

The bulk of the equipment used can also play a role in what methods are more advantageous. For example, the pipette apparatus used for the pipette method is typically attached to the wall or stand of some kind. Due to the fact that the cylinders can not be disturbed in the hours leading up to the subsamples being taken, only the number of cylinders that fit within the width of the apparatus can be run. This limits the number of samples that can be done in the day simply because of the limited reach of the apparatus. The hydrometer method has an advantage in this regard as it is more mobile and additional samples can be run. While there is still a limit to the number of samples that can run at a time due to the collection of samples occurring at specific intervals and perhaps limited counterspace, the limit put upon the samples does not come from the bulk of the equipment with this method for analysis.

As with most scientific processes, the cost of equipment also plays a role in the determination of which method would be best. In comparison to other methods used for particle size analysis, both the pipette method and hydrometer method are relatively inexpensive. That's not to say that they do not cost a significant amount of money, it just happens to be less than the expense of other methodologies. Both methods require the more basic equipment of 1 L cylinders, sieve sets, shakers, etc.. While these do add to the expenditure required, it does not play a role in which method has an advantage in cost. A pipette apparatus purchased from Eijkelkamp North America costs approximately $1,733. A hydrometer purchased from Fisherbrand costs approximately $50.30. These price points are far from one another and leave the hydrometer method being more advantageous if an individual is looking for a more cost-efficient method of carrying out Particle Size Analysis.

Based upon an experiment completed in 1985, the methods are more reliable depending upon the samples that are being analyzed. Those with a higher percentage of fine particles were more reliably determined through the hydrometer method, as determined through an average standard deviation (Coates & Hulse, 1985). The pipette method more reliably determined samples with a higher percentage of coarse particles, as determined through an average standard deviation (Coates & Hulse, 1985). If all types of soils are being analyzed, the pipette method had the overall smallest average standard deviations across all samples (Coates & Hulse, 1985). While the results for each were only slightly better or worse, it is an aspect to consider.

Conclusion

The hydrometer and pipette methods each display distinct advantages and disadvantages. In the end the determining factor of what is best for research comes down to the researcher and the resources available. For those looking for the most cost-effective manner to complete Particle Size Analysis, the hydrometer method holds a distinct advantage in this regard. Those looking for a method requiring less time would look to the pipette method. An important aspect to consider is the type of soil samples that will be analyzed using the method chosen. As mentioned above those with a higher percentage of fine particles present are better analyzed through the hydrometer method, with the pipette holding an advantage for those samples containing a higher percentage of coarse particles. If samples are unknown or the samples being run vary greatly in their composition, the pipette method had more accurate results across all sample types, but either method would yield appropriate results. Both methods will supply appropriate results in the individual and will help in the process to determining the soil composition of the sample being analyzed. The hydrometer and pipette methods rely upon the same basic principles for the completion of analysis and therefore sample analyzed through either method should yield results that are able to be compared to those obtained through the other method.

References:

Black, C. A. (1965). *Methods of soil analysis*. Madison, Wis: American Society of Agronomy

Bouyoucos, G. (1927). The Hydrometer as a New Method for the Mechanical Analysis of Soils. *Soil Science.*, *23*(5), 343-354.

Bouyoucos, G. (1962). Hydrometer Method Improved for Making Particle Size Analyses of Soils. *Agronomy Journal*, *54*(5), 464-465.

Coates, G. F., & Hulse, C. A. (1985). A comparison of four methods of size analysis of fine grained sediments. *New Zealand Journal of Geology and Geophysics, 28*(2), 369-380.

Dane, J., Topp, G., & Campbell, G. (2002). *Methods of Soil Analysis. Part 4, Physical Methods.* Madison, Wis.: Soil Science Society of America.

Garcia-Gaines, R. A., & Frankenstein, S. (2015). *USCS and the USDA Soil Classification System: Development of a Mapping Scheme* (Rep.). Vicksburg, MS: US Army Engineer Research and Development Center. Retrieved October, 2018.

Jury, W., & Horton, R. (2004). *Soil Physics* (6th ed.). Hoboken, N.J.: J. Wiley.

Klute, A. (1986). *Methods of Soil Analysis. Part 1. Physical and Mineralogical Methods* (2nd Ed.). Madison, Wis.: American Society of Agronomy, Inc. & Soil Science Society of America, Inc.

Mirosław Orzechowski, Sławomir Smó;lczyński, Jacek Długosz, Paweł Pozniak, Orzechowski, M., Smó;lczyński, S., … Pozniak, P. (2014). Measurements of Texture of Soils Formed from Glaciolimnic Sediments by Areometric Method, Pipette Method and Laser Diffraction Method. *Soil Science Annual.*, *65*(2), 72–79.

Väderstad Group. (n.d.). Characteristics of different soil types. Retrieved from https://www.vaderstad.com/en/know-how/basic-agronomy/soil-basics/characteristics-of different-soil-types/